



165593

October 19, 2001

Mr. Mike Ribordy
U.S. EPA – Region 5
77 West Jackson Boulevard (SR-6J)
Chicago, IL 60604-3590

RE: Sauget Area 2 Sites

Deliver by Overnight Mail

Dear Mike,

Enclosed please find one additional Standard Operating Procedure (SOP) for your review in accordance with our discussions via conference call last week (10/9/01) on this topic. The SOP that has been attached includes the following:

Severn Trent Laboratories, On-Site Technologies Division (mobile laboratory for soil screening)

- SOP OSMM1012.R1 Heavy Metals by XRF

Severn Trent (the On-Site Technologies Division) has modified the SOP for metals by X-Ray refraction (XRF) to account for site-specific calibration procedures, based on the discussions held during the conference call on this topic.

Please do not hesitate to call Steve Smith if you have any questions on this information.

Sincerely,

Robert B. Veenstra
Senior Program Manager

Cc: Steve Smith, SA2SG Project Manager

Effective Date: 08/21/01

STL On-Site Technologies STANDARD OPERATING PROCEDURE

TITLE : Heavy Metals in Soil by Energy Dispersive X-Ray Fluorescence Spectroscopy

SUPERSEDES : (NONE)

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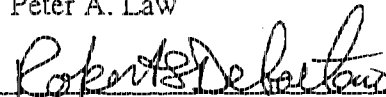
Division Manager:



Peter A. Law

8/30/01
Date

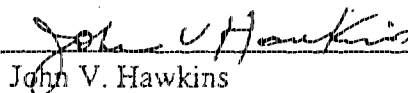
QA Manager:



Robert S. DeGaetano

8/22/01
Date

Technical Director:



John V. Hawkins

8/29/01
Date

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1.0 SCOPE AND APPLICATION

- 1.1 The purpose of this SOP is to outline the techniques for determining the concentration of certain metals in soil or sediment samples. Typical reporting levels are as follows:

Table 1 - Reporting Limits for Select Elements by EDXRF

Element	Spectrace 6000 (ppm)
Antimony	10
Arsenic	10*
Barium	10
Cadmium	10
Chromium	20*
Copper	10
Lead	10
Mercury	10
Nickel	10
Selenium	10
Silver	10
Thallium	10
Zinc	10

* Subject to interferences -- see section 4

- 1.2 This method is a screening technique and is based on EPA method 6200. Off-site confirmation using approved techniques is suggested to compliment any investigation.
- 1.3 It is the policy of STL and of the On-Site Technologies Division to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the STL Policy Statement on Business Ethics and Conduct.

2.0 SUMMARY OF METHOD

- 2.1 Samples are prepared by a combination of techniques including drying, sieving and pulverizing prior to analysis. Once prepared, approximately five grams of the sample is placed into a X-ray sample cup and covered with Prolene® X-ray film. The sample is analyzed under preset energy conditions for the elements of concern. The software is programmed to calculate final concentrations by "Fundamental Parameters" or by a least squares fit to 'site-specific' calibration data. The software algorithms correct for matrix effects, and calculate final concentrations by an iterative process.

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- 2.2 Background - The Field Portable X-Ray Fluorescence (FPXRF) technologies described in this method use sealed radioisotope sources to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. As a result, a rearrangement of electrons occurs resulting in emission of x-rays characteristic of the given atom. It is this emission of x-rays, or fluorescence, that is measured for both reference standards and samples to obtain metals concentrations.
- 2.3 Analysts must be appropriately trained by an experienced operator and demonstrate acceptable proficiency prior to conducting this analysis without direct supervision.

3.0 DEFINITIONS

- 3.1 There are many definitions used within the laboratory that may be generic to all laboratory analyses or more specific for certain methods. For the most recent terms and definitions used within the laboratory, refer to the STL-OST Laboratory Quality Manual.

4.0 INTERFERENCES

- 4.1 The heterogeneity of the sample has a large impact on comparability of results with confirmatory analysis by other spectroscopic techniques such as ICP and GFAA. Consequently, it is imperative to homogenize the sample as best as possible.
- 4.2 Moisture content may affect the accuracy of analysis of soil and sediment samples. This effect may be minimized by drying the samples in a convection oven prior to analysis. However, if mercury is to be tested, the sample should be dried at ambient temperature.
- 4.3 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the sample distance between the window and each sample. For best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.
- 4.4 Chemical matrix effects result from differences in the concentrations of interfering elements. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients or by using a site-specific calibration standard (SSCS). Examples of interfering elements are as follows:
- Iron (Fe) tends to absorb Copper (Cu) x-rays and enhance Chromium (Cr)
 - High concentrations of Lead (Pb) may result in no Arsenic (As) being reported

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5.0 SAFETY

- 5.1 It is strongly recommended that analysts treat all samples as if they are hazardous and take all appropriate safety precautions. Analysts shall wear appropriate personal protective equipment including lab coats, safety glasses with side shields and impervious gloves (Nitrile, eg. Sol-Vex).
- 5.2 Proper precautions should be taken when formulating new standards, or handling suspected high concentration samples.
- 5.3 For long term projects, radiation monitoring equipment should be used.
- 5.4 Material Safety Data Sheets for all chemicals used in the operation are present in the laboratory for immediate access.

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 6.1 Samples should be collected in glass jars and stored at 4 ± 2 °C until analysis.
- 6.2 Sample can be tested up to 6 months after collection for all metals except mercury which must be analyzed within 28 days of collection.

7.0 EQUIPMENT AND MATERIALS

- 7.1 Spectrace Instruments Model 6000 Field Transportable EDXRF Analyzer with software
- 7.2 Spectrace Instruments Model 9000 Field Portable EDXRF Analyzer with software.
- 7.3 Prolene® X-ray film, Chemplex Industries 1/6mil or equivalent.
- 7.4 Chemplex polyethylene X-ray sample cups.
- 7.5 Mortar and Pestle suitable for grinding environmental soils.
- 7.6 Spex Model 8000 Roller mill with tungsten carbide vial set and balls.
- 7.7 Drying trays: polyethylene or paper.
- 7.8 Drying oven: microwave oven or temperature controllable convection oven.
- 7.9 Stainless steel spatulas.

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7.10 Scintillation vials: 20ml with plastic caps.

7.11 Manufacturer's Operations Manual

8.0 REAGENTS AND STANDARDS

8.1 NIST Certified Soil Reference Material (SRM): purchased directly from NIST, Gaithersburg, Maryland. For Fundamental Parameters, STL-OST calibrates with NIST SRM 2704, 2709, 2711. Specific sites may require purchase of a SRM with a particular elemental composition. Consult the NIST catalog for possible SRM.

9.0 PROCEDURES

9.1 Set-Up

9.1.1 The Spectrace 6000 must have power to the ion pump at all times. This pump maintains a vacuum on the X-ray source. While connected to shore power, the onboard Nicad battery is constantly charged and will supply approximately three days of power to the ion pump. If longer transport time is required, connect a full-charged 12V external battery to the ion pump leads.

9.1.2 Arrange the components with the printer and computer to the left and the card cage to the right of the head unit. Turn all components on. If the instrument begins to beep, perform trouble-shooting activities and refer to operator's manual to correct the problem.

9.1.3 If applicable, turn the defrost switch off, and the cooler switch on to initiate detector cooling. Wait at least one hour for detector to reach operating temperature.

9.2 Energy Calibration

9.2.1 Prior to calibration and sample analysis, the analyst must run an energy calibration. This procedure resets the instrument gain, resolution, and peak offset relative to a pure copper standard, which compensates for drift due to changing temperature conditions.

9.2.2 Be sure that the computer is set to the correct directory. Place the copper disk in the sample chamber and press F7 to initiate the energy calibration. The calibration takes approximately seven minutes. Print out a copy of the instrument setting listed on the PHA status page.

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- 9.2.3 Perform an energy calibration at the beginning of each day, once every twenty samples, and after significant changes in ambient environmental conditions.
- 9.2.4 Instrument gain settings should not change appreciably between consecutive energy calibrations. If the gain changes by more than +/- 15%, check instrument hardware (especially filter solenoids) and software for problems and repeat energy calibration.

9.3 Initial Calibration

- 9.3.1 Setup instrument conditions that will allow for 50% dead time in all energy levels (low, mid, high). It is possible to customize the method for a particular analyte by varying energy level livetimes, tube voltage and current. Varying the livetime will affect the sample throughput. Once the instrument conditions have been set for a particular method, they must remain the same for all subsequent calibration and sample analysis events. An example of instrument settings for each of the three energy levels is shown in Appendix A.
- 9.3.2 Initial calibration is prepared at a minimum of three levels. Use three NIST SRM's that contain the elements of interest. Calibration is performed using a fundamental parameters algorithm with NIST SRM's 2704, 2709, 2711. A project-specific linear calibration may be performed for single elements using site-specific standards. These standards are analyzed by ICP or graphite furnace prior to the start of the project and the values obtained are used in the XRF calibration table.
- 9.3.3 Set up the calibration table for all elements of interest. Enter the certified values from the NIST standard documentation. enter non-certified values in parenthesis. If a site-specific calibration is used, enter the values provided by the ICAP or AA analysis as certified values into the calibration table.
- 9.3.4 Change directories using the F5 soft key into the appropriate method directory. Highlight the calibration option line for the current method. Press F1 soft key to begin calibration. Follow screen instructions and insert appropriate SRM into the sample chamber. Repeat for all three SRM's.
- 9.3.5 After the initial calibration is completed, insert an appropriate SRM to be used as the laboratory control standard (LCS). The SRM should contain the analytes of interest, however it may not be possible to obtain a SRM with all analytes of interest at appropriate concentrations.

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- 9.4 Continuing calibration is done at a sample frequency of 20% or when malfunction of the instrument is suspected. Calibration check is done by analyzing one of the three SRM standards used for calibration. If the result varies from the true value by more than 30% for the analytes of interest, a new calibration must be prepared. The variation is calculated as a Percent Difference (PD):

$$\text{Percent Difference} = \frac{R1 - R2}{R1} \times 100$$

where:

R1= true value

R2= the measured value.

- 9.4.1 If the certified value of the SRM is within five times the reporting limit, the PD criteria will not apply. If the continuing calibration fails for the compounds of interest, check the hardware and software settings for the instruments, especially the filter solenoids. Be sure that the correct method directory is in use.

9.5 Soil Preparation

- 9.5.1 Remove foreign debris such as sticks and leaves, if present and screen samples through a 10 mesh sieve.
- 9.5.2 Dry all samples prior to quantitative analysis. Drying technique may include drying by air, microwave oven, or convection oven. If mercury is an analyte of interest, excessive drying temperature can cause it to volatilize. It is recommended to not exceed 40°C when drying samples containing mercury or other volatile metals.
- 9.5.3 Pulverize approximately 10g of the sieved sample by grinding with a mortar and pestle, or equivalent. The sample should be ground to a rock flour consistency (attempt to grind to same consistency as SRM standards).
- 9.5.4 Place approximately 5g of dried and pulverized sample into an XRF sample cup. Cover the cup with a square piece of Prolene® X-ray film, and secure in place with the plastic rings. Inspect the X-ray film to see that it is free of wrinkles. Wrinkles must be removed prior to analysis.

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9.6 Sample Analysis

- 9.6.1 Sample analysis may begin following successful energy calibration, calibration check, and laboratory control standard analysis.
- 9.6.2 Analyze samples under same energy conditions as calibration standards.
- 9.6.3 Place sample in XRF sample chamber. Using the cursor, highlight the appropriate analysis method and press the F1 soft key. Enter sample ID at the prompt and press spacebar to start analysis. The instrument will analyze all energy levels programmed into the method and print out a hard copy at the end of the analysis. Report calculated values from printout for elements of interest.
- 9.6.4 The analyst must determine whether low level hits for elements of interest are valid. The following test may be used to determine whether a peak is above background signal noise:

$$\text{peak counts} > 3 * \text{background check counts}$$

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10.0 QUALITY CONTROL

10.1 **Energy Calibration:** See Section 9.2

10.2 **Laboratory Control Standard (LCS):** An NIST SRM may be run as an LCS. The acceptance criteria is +/- 30% of the certified value. Consult the NIST catalog for available SRMs. The SRM should contain the element of interest at an appropriate level. For multiple elements, it may not be possible to find a LCS with all elements of interest.

10.3 **A sample duplicate** is to be analyzed with each batch of twenty samples. The relative percent difference between the initial and duplicate result must be $\leq 35\%$. Calculate the RPD according to the following formula:

$$\text{RPD} = \frac{\text{ABS } (C_1 - C_2)}{(C_1 + C_2)/2} \times 100$$

where:

C_1 = Concentration from initial sample

C_2 = Concentration from duplicate sample

10.3.1 If the compound of interest is at a concentration within five times the reporting limit, the RPD criteria will not apply.

10.4 **Corrective Action:** Corrective action must be performed when quality control data exceeds acceptance criteria. Activities performed, verification of successful corrective actions and affected samples should be recorded on the Quality Assessment Deviation Form provided as Appendix B to this document.

10.5 **A detection limit study** must be performed initially and after any significant changes to the methodology or when significant instrument maintenance is required. To perform the MDL, obtain an SRM containing the elements of interest at the appropriate levels and generate a minimum of seven sequential measurements conducted over three non-consecutive days.

11.0 CALCULATIONS

11.1 Results are calculated by the manufacturer's software.

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12.0 DATA REDUCTION and REVIEW

- 12.1 Hardcopies of all instrument calibration, calibration verification and daily analytical sequence raw data shall be assembled into "packages" for review.
- 12.2 Analysts are responsible for reviewing the packages to evaluate the accuracy of transcriptions, the instrument's performance, and the calculated results. Section 10 details the acceptance criteria and corrective actions for instrument and batch QA/QC.
- 12.3 All necessary documentation to support corrective actions performed shall be included with the packages and available for review.
- 12.4 Preliminary results may be reported to the client at this time.

13.0 REPORTING OF RESULTS

- 13.1 Prior to generating a final report of results, the data is reviewed a second time. In addition to the method review performed by the first chemist, the second review includes a verification of any project/client specific criteria and a sensibility check.
- 13.2 If for any reason there is a question regarding the technical soundness of the data, it is brought directly to the attention of the Technical Director and/or Quality Assurance Manager.
- 13.3 After completing the secondary review, the final results are reported to the client in hardcopy form and/or electronic deliverable, if requested.

14.0 POLLUTION PREVENTION

- 14.1 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W. Washington D.C. 20036, (202) 872-4477.

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15.0 WASTE MANAGEMENT

- 15.1 No solvents or acid waste is generated.
- 15.2 Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.
- 15.3 Refer to the Laboratory Sample and Waste Disposal plan.
- 15.4 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16.0 SUPPLEMENTAL DOCUMENTS – N/A

17.0 REFERENCES

- 17.1 EPA Method 6200, January 1998

18.0 SUBSTANTIVE REVISIONS

- 18.1 Original issue – 05/15/01
- 18.2 Revision #1 – 08/21/01

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Appendix A

Parameters for Low, Mid, High Energy Conditions: Spectrace 6000

Parameter	Low	Mid	High
Tube Voltage	13KV	23KV	50KV
Tube Current	0.35mA	0.35mA	0.35mA
Filter	Aluminum	Thin Palladium	Copper
Livetime*	50-200sec	50-200sec	50-100sec
Preset Count	0 K	0 K	0 K
Max Energy	10 KeV	20 KeV	40 KeV
Atmosphere	Air	Air	Air
Warmup	0 sec	0 sec	0 sec
Elements of Interest K α	K, Ca, Ti, V, Cr, Mn	Fe, Co, Ni, Cu, Zn, As, Se	Ag, Cd, Sb, Ba
Elements of Interest L α		Pb, Hg	
Elements of Interest *FP	Fe	Mn, Br, Rb, Sr, Y, Zr, W	Sn

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Appendix B
Quality Assessment Deviation Form

DATE: _____

DATE OF ANALYSIS: _____

TIME: _____

PROJECT #: _____

ANALYST: _____

SDG #: _____

SAMPLES AFFECTED BY THIS DEVIATION

LAB SAMPLE ID	LAB SAMPLE ID	LAB SAMPLE ID	LAB SAMPLE ID

QC SAMPLE

QC DEVIATION

LIMITS

CORR. ACTION

COMMENTS: _____

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**SEVERN TRENT LABORATORIES
ON-SITE TECHNOLOGIES (OST) DIVISION**

APPENDIX TO STANDARD OPERATING PROCEDURE OSMM1012

TITLE: Heavy Metals in Soil by Energy Dispersive X-Ray Fluorescence Spectroscopy

Approvals and Signatures

Division Manager:

Peter A. Law

Date

QA Manager:

Robert S. DeGaetano

Date

Technical Director:

John V. Hawkins

Date

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1.0 SCOPE AND APPLICATION

- 1.1 This appendix to SOP OSMM1012 presents the additional procedures and modifications of original procedures for the determination of site-specific calibration standards (SSCS) for XRF analysis. All aspects of the analysis not discussed here are performed as described in the main body of referenced SOP.

2.0 SUMMARY OF THE METHOD

- 2.1 Soil samples are collected from the site under investigation and analyzed by Inductively Coupled Plasma Spectroscopy (ICP) and/or Graphite Furnace Atomic Absorption (GFAA). The metals concentrations obtained are then compared to the values generated using X-Ray Fluorescence (XRF) for the same samples. Calibration curves for each element of concern are then prepared using the concentrations as determined by ICP and/or GFAA and the associated responses from the XRF analysis. These calibration curves are then used to calculate metals concentrations for samples taken from the site.

3.0 SAMPLE COLLECTION AND PRESERVATION

- 3.1 Samples should be collected in glass jars and stored at 4 ± 2 °C until analysis.
- 3.2 Samples can be tested up to 6 months after collection for all metals except mercury, which must be analyzed within 28 days of collection.
- 3.3 Sufficient quantity and sources must be collected from the site to yield multiple concentration levels for each element of concern as well as the associated interfering element(s).

4.0 PROCEDURES

4.1 Site Specific Calibration Standard Preparation

- 4.1.1 The SSCS samples collected in the field must be oven-dried at < 150 °C for 2 to 4 hours to remove moisture.
- 4.1.2 The entire sample should be spread out in a drying pan and clumps broken up with a stainless steel spoon.
- 4.1.2.1 If mercury is to be analyzed, a separate sample must be air dried until the moisture content is < 20 %, as heating may volatilize the mercury.
- 4.1.3 After drying, all large organic debris and non-representative material (sticks, twigs, leaves, roots insects, asphalt, rocks, etc.) are removed and the sample is transferred to a mortar and ground with a pestle to a uniform consistency.

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- 4.1.4 The dried and ground sample is then sieved through a No. 60 (250 μ m) mesh stainless steel sieve. At no time should the material be forced through the sieve. Pebbles and organic matter remaining on the sieve should be discarded.
- 4.1.5 The under-sieve fraction of the material constitutes the sample.
- 4.1.6 Homogenize the sieved sample and fill an XRF sample cup. Transfer the remainder of the sample back into the original container and return it to storage until analysis by ICP and/or GFAA.
- 4.1.7 Analyze the sample according to procedures found in SOP OSMM1012 and record the intensities of the elements of concern.
- 4.1.8 Repeat for all SSCS soil samples.
- 4.1.9 For each SSCS sample, analyze the ICP/GFAA portion in triplicate and average the concentration values to obtain a "certified" value.
- 4.1.10 Generate calibration curves for each element of concern using the intensity values from the XFR analysis and the "certified" concentration results from the ICP/GFAA analyses.

Note – these calibration procedures are used in place of for the ones identified in Section 9.3 of SOP OSMM1012

- 4.1.11 **If all elements of concern are not represented in the samples collected from the site, substitute values generated from the XRF and ICP/GFAA analyses of certified reference material soil samples.**

4.2 Sample analysis

- 4.2.1 Dry, sieve and homogenize the field sample as described above.
- 4.2.2 Analyze the sample by XRF according to procedures outlined in SOP OSMM1012.
- 4.2.3 Calculate the elemental concentrations using the intensity values from the XRF and the calibration curves generated above (see calculation section in this SOP).

5.0 QUALITY CONTROL

- 5.1 The Quality Control is essentially the same as discussed in SOP OSMM1012. Modifications to the acceptance criteria may be made at the time of project inception due to matrix interferences and or data quality objectives for the project. These modifications, however, must be made in writing and communicated to the client for approval.

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6.0 CALCULATIONS

1. **Linear Regression** – plot intensity of SSCS as determined by XRF analysis (y) versus “certified” concentrations as determined by ICP/GFAA (x)
 - $Y = M * X + B$ (where M = slope)
 - Determine correlation coefficient (r) – result for r must be > 0.990
 - To obtain sample concentration, enter intensity for elements and solve for x.
2. **Weighted Linear Least Squares Regression**– plot intensity of SSCS as determined by XRF analysis (y) versus “certified” concentrations as determined by ICP/GFAA (x)
 - $Y = M * 1/X + B$ or $Y = M * 1/X^2 = B$
 - Determine correlation coefficient (r) – result for r must be > 0.990
 - To obtain sample concentration, enter intensity for elements and solve for x.